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Original Article

INVESTIGATION OF DENSIFICATION BEHAVIOUR ON YTTRIUM OXIDE REINFORCED TI-6AL-4V NANO-COMPOSITE THROUGH POWDER METALLURGY

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ABSTRACT

Titanium based alloys are attracted in many applications due to its excellent properties. But it is not suitable for high temperature components due to poor performance at elevated temperatures. In this paper, a new attempt is done to improve its high temperature performance by adding yttrium oxide (Y_2O_3) as reinforcement in a titanium alloy matrix. Yttrium oxide reinforced Ti-6Al-4V nano-composite was manufactured through powder metallurgy technique. Yttrium oxide was added in a range of 1-3% with matrix metal. The compaction pressure of 560 MPa was determined by using powder compression techniques. The composite preforms was made by cold compaction and green compacts were sintered through pressureless sintering process at a temperature range of 1200-1400^oC to improve the densification behaviour. Densities of preforms were measured by using analytical and Archimedes' principles to determine the densification behaviour of nano-composite. The scanning electron microscopy (SEM) and X-ray diffraction (XRD) were used to confirm the distribution of reinforcement with matrix metal. The nano-composite prepared by adding 2% of yttrium oxide provides higher densification behaviour compared to other additions of reinforcements.

KEY WORDS: Powder, Sintering, Densification, Compacts & Reinforcements

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INTRODUCTION

Metal matrix composites (MMC) are more attracted by the manufacturing industries due to its superior mechanical and thermal properties [1, 2]. The titanium alloy of Ti–6Al–4V is used in the applications in aerospace and biomedical applications. This type of alloys provides excellent mechanical properties like high static and fatigue strength, low density, high specific strength, high fracture toughness, excellent corrosion resistance even at elevated temperatures [3-9].

Researchers [10-13] have proved that the matrix of titanium and titanium alloys properties are potentially improved when the ceramic particles reinforced in it. Titanium matrix has been reinforced with TiB, TiC, Al_2O_3, SiC, TiB_2 etc. and investigated by many researchers [14].

Ti6Al4V is considered as the reasonable candidate due to outstanding combination of mechanical properties for various service conditions for the modern industrial components. The powder metallurgy route was used to fabricate the titanium composite to avail the superior and isotropic properties with low cost [15-19]. But, the use of these alloys is very limited due to poor oxidation resistance under elevated temperatures [20, 21]. The components of powder metallurgy are observed with lesser density compared to wrought materials, but which fulfills the required parameters for the industrial applications. The strength of the P/M components can be enhanced through the strengthening mechanism such as cold upsetting processes [22, 23]. Titanium alloys metal matrix composites manufactured through conventional powder metallurgy reveal an excellent combination of improved properties of toughness and strength, which disables the extreme brittleness of titanium alloys. Frary etl [24] fabricated a composite of tungsten reinforced in the Ti6Al4V matrix through powder metallurgy and proved that the improved mechanical properties of strength and hardness by losing minimum ductility. Several methods are available to compact metal powders such as uniaxial dies compaction [25, 26], injection molding [27,28] and isostatic compaction [29, 30] under either room or elevated temperatures. The main aim of this paper is to explain the conventional method of powder metallurgy technique and the influence of densification processes of Ti6Al4V/Y_2O_3 metal matrix composite. The composite and alloy preforms were manufactured through processes such as the blending of powders, cold compaction of powders to produce green compacts and pressureless sintering under controlled atmosphere. The compaction pressure was measured through uniaxial compression test and the same pressure was used to produce green compacts.

MATERIALS AND METHODS

The Ti6Al4V matrix metal powder having 98.0% purity of -325 meshes size and reinforcements as Yttrium Oxide having 99.995% purity of 40 nm particle size were used to prepare a nano-composite of Ti6Al4V/Y_2O_3 . The chemical elements of matrix and reinforcement materials are given in the Table 1 and 2.

Table 1: Chemical Element in Ti-6al-4v (Powder)

Chemical	H	Ni	Si	Mg	Al	O	Cl	Fe	Mn	V	Ti
Content in %	0.018	0.005	0.003	0.08	6.10	0.20	0.04	0.12	0.04	3.81	Balance

Table 2: Chemical Elements in Y_2o_3 (Powder)

Chemical	Zn	La	Si	Mg	Al	Ca	Fe	Mn	Y_2O_3
Content in %	0.0005	0.001	0.001	0.0005	0.0005	0.001	0.001	0.0001	Balance

Densification of powders is mostly depending on the applied pressure which can be explained by using density–pressure curves. This curve has been extensively used to predict and study the compaction behavior of powders [31, 32]. The compaction pressure was estimated to make compaction of composite. The measured quantity of metal powder was poured in the compaction die and load applied through the punch. The die and punch surfaces were lubricated before pouring the powder by using molybdenum di-sulphide to avoid the friction between powder compacts and wall surfaces of the press tool. The load was gradually increased to the powder through the punch and reduction of height of the powder was noted down while the load was applied to the particular interval. The rate of reduction in height of the powder was diminished with increase in pressure beyond the certain limit. The details of the computational model can be found elsewhere [33, 34]. Figure 1 shows that the high rate of increase in density was observed at the initial stage of loading, but it was started to reduce the further load increases. There was no reduction in height observed after reaching certain applied pressure, that is constant density or no densification occurs beyond the maximum pressure as shown in Figure 1,.

The compaction pressure was measured using the 100 ton capacity hydraulic press.

The Ti6Al4V/Y_2O_3 metal matrix composites were prepared through powder metallurgy technique. Manufacturing of composites consists of blending, compaction and sintering processes. The required quantity of matrix and reinforcement material powders was mixed uniformly to avoid heterogeneous properties of composites. Ball mill was used to mix the powders for 1 hour. The blended powders were used to prepare alloy and composite preforms by cold compaction process. the known amount of powders was poured into a hollow cylindrical die which available in a hydraulic press having a capacity of 100 tons. The bottom side of the die was covered with bottom punch to arrest the powder particles inside of the die. The surfaces of die, bottom punch and top punch were lubricated with molybdenum di-sulphide to avoid friction between powder and surfaces which also helped to eject preforms smoothly from the die. The top punch was inserted in the top side of the die and compaction pressure of 560 MPa was applied gradually through the ram of a hydraulic press. The green compacted preforms were carefully ejected to avoid damage. The Ti6Al4V composite of having 1, 2 and 3% of yttrium oxide was fabricated with similar procedures. The pure Ti6Al4V sample was prepared to compare the results with composites. The green density was evaluated through analytical and Archimedes' principle. The uniaxial compaction process mechanism can be explained in three stages such as initial, intermediate and final stages of compaction. When the load started to apply at the earliest stage of compaction, the gap between the particles gets reduced due to the rearrangement mechanism under low load. The packing density of particles was increased due to the initial stage of compaction. If a load is increased further, powder particles are deformed plastically during the intermediate stage of compaction. The maximum load was applied at the final stage of compaction. The density and density distribution of green compacts depend on the certain parameters such as shape and size of the particles, coefficient of friction, compaction pressure and temperature, work hardening and ejection pressure.

The green compacts were sintered in a helium gas (controlled atmosphere) filled sintering furnace at temperatures of 1200^{o}C, 1300^{o}C and 1400^{o}C for 2 hours. The controlled atmosphere was used to do sintering process for eliminating the process of oxidation during the process. The heating process was done at 15^{o}C/min and cooling was done at 10^{o}C/min The sintered samples were machined to the required size and measured the density. The same procedure was followed to manufacture the remaining samples. The measured values of densities were tabulated.

The mechanism of sintering is also explained in the three stages such as initial, intermediate and final stages of the sintering process. At the initial stage of heating, neck formation taken place between the particles. But the neck sizes were considerably small due to the lower temperature. During the intermediate stage, neck size grows due to the increase in temperature, which moves the particles closer and reduced the porosity. The entire pores disappear at the final stage of sintering processes due to the higher movement of the particles and grain boundary development along the pores.

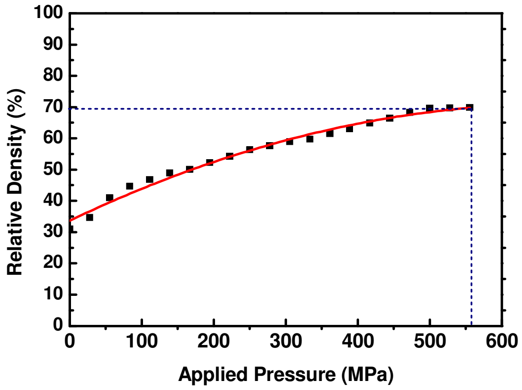

Figure 1: Relation Between Relative Density and Applied Pressure on Metal ti6al4v Powder

RESULTS AND DISCUSSIONS

The sintered composites are taken thru scanning electron microscopy (SEM) and X-ray diffraction to confirm the presence of Ti6Al4V and Y_2O_3 particles. The samples are prepared as per the standard specimen preparation method for metallographic examination. The emery papers having grit size of 320, 600, 800 and 1200 were used to grind the composite surfaces. The ground sintered preforms were polished in a mechanical polishing machine by applying diamond paste having 1 and 5 micrometer particle sizes on a rotating disc. The polished composite preforms were chemically etched by using solution contains water, hydrochloric acid and hydrofluoric acid in proportions as given in the ASTM standard. Figures 2 (a) -2 (d) indicate the SEM image that reveals the particles of Ti6AL4V and Y_2O_3. Figure 2 (a) shows the grain of Ti6Al4V and clear grain boundaries. Figures 2 (b) – (d) show the microstructures of composite having 1, 2 and 3% Y_2O_3 respectively. Figure 2 (d) is shown that the reinforcement particles have not been distributed uniformly, so that the densification decreases in this composite. Figure 3 displays the distribution of reinforcement particle in the matrix of titanium alloy. It is clearly indicated that the Y_2O_3 particles are evenly distributed in the Ti6Al4V matrix phase.

The apparent and tap densities of both matrix and reinforcement metal powders were calculated and tabulated in Table 3.

Table 3: Apparent and Tap Densities of Powders

Name of Metal Powder	Apparent Density in g/cm^3	Tap Density in g/cm^3
Ti6Al4V(matrix metal)	1.3906	1.9307
Y_2O_3(Reinforcement material)	0.3851	0.4812

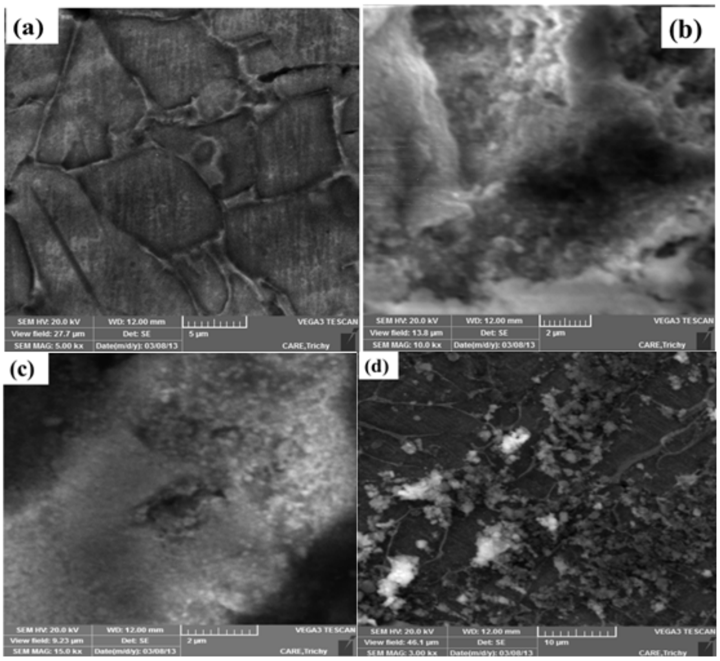

Figure 2: SEM Images of Sintered (a) Ti6Al4V Alloy (b) 1% Y_2O_3 Composite (c) 2% Y_2O_3 Composite (d) 3% Y_2O_3 Composite

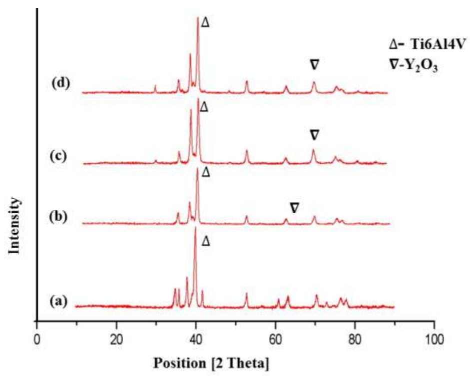

Figure 3: XRD Profile of (a) Titanium Alloy (Ti6Al4V) (b) Composite with 1% Y_2O_3 (c) Composite with 2% Y_2O_3 (d) Composite with 3% Y_2O_3

The theoretical density was determined by using the rule of mixture for various composites to compare the density values of green compacts and sintering composites. The density of sintered composites was calculated using analytical and Archimedes' principle method. Figure 4 shows in the density variation in different composition of yttrium oxide in the matrix of Ti6Al4V.

The theoretical density is also indicated in Figure 4 to compare the sintering density at various temperatures. The theoretical density of composite slightly increases with the addition of yttrium oxide. The sintered densities of various composites are different and lower values compared to the theoretical densities. The highest sintered density is obtained in the pure Ti6Al4V compared to the composite materials (1%, 2% and 3% of Y_2O_3) in all sintered temperatures (1200°C, 1300°C and 1400°C). The increase in sintering temperature influences the density towards the higher value. The composites of 2% of Yttrium Oxide have a higher sintered density compared to 1% and 3%. The rate of change of density

increases when the reinforcement percentage increases from 1% to 2%, but this trend is changed after 2% for the all sintering temperatures. The rate of change of the decrease in density after 2% is lower at a 1300°C sintering temperature compared to 1200°C and 1400°C. The composite having 2% yttrium Oxide has better densification compared to 1% and 3%.

Figure 5 shows that relation between the density and amount of reinforcements with respect to different sintering temperatures. The highest density is reached in pure titanium alloy under different sintering temperatures, but 1400°C gives the higher density value. The amount of reinforcement increases in the Ti6Al4V matrix, densification starts to decrease upto 1% of Y_2O_3. The reinforcement amount increases from 1 to 2%, densification starts to increase up to 2% till the 3% of addition leads to decrease the densification. The densification of all composites is varied with sintering temperatures, but the 1400°C is dominating the other sintering temperatures. The highest density is observed in the composite having 2% reinforcement irrespective of sintered temperatures.

Figure 6 shows the relative density values of pure Ti6Al4V and composites for various sintering temperatures, namely 1200°C, 1300°C and 1400°C. The highest relative density is obtained in the pure Ti6Al4V alloy. The sintering temperature increases from 1200°C to 1300°C observed that the density increases, even though the further increase in temperature influences the decrease in density values up to 1400°C in the alloy. The composite samples are observed that the rise in sintering temperature gradually increases the density values from 1200°C to 1400°C. The highest density is found in the Ti6Al4V/2% Y_2O_3 composite sintered at 1400°C.

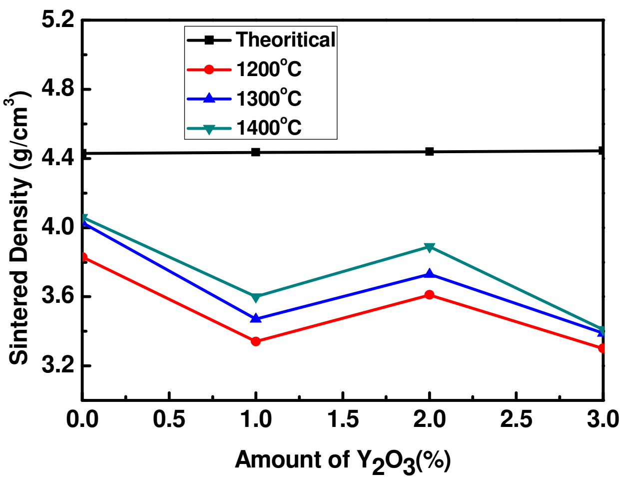

Figure 4: Density Variation with Amount of Reinforcements after Sintering

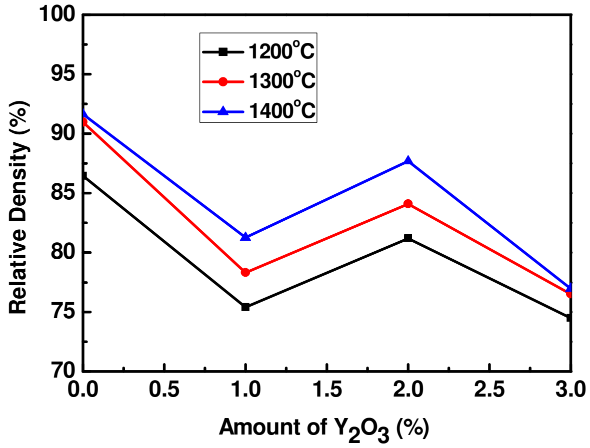

Figure 5: Density Variations with Different Amount of Reinforcements

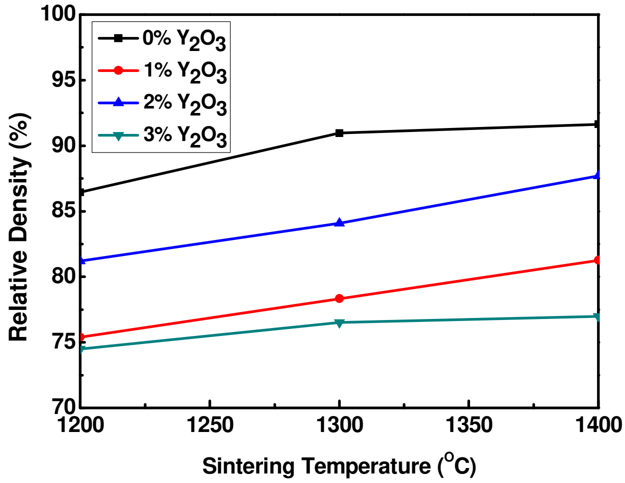

Figure 6: Relative Densities of Pure Ti6Al4V and Composites for Various Sintering Temperatures

CONCLUSIONS

Titanium alloy composite (Ti6Al4V/Y_2O_3) was prepared through powder metallurgy route and the following remarkable conclusions are made:

The compaction pressure was estimated as 560 MPa under the uniaxial compression. The composite is having 1 and 2% of Y_2O_3 was distributed as fine particle in the matrix of Ti6Al4V rather than 3% of Y_2O_3. Accurate density values were measured through Archimedes principle method than the conventional method. The highest densification was achieved in the alloy rather than composite samples. Green compacts strength was obtained due to the mechanism of cold welding and mechanical interlocking of powder particles at the final stage of compaction. The amount of reinforcement increases in the matrix phase, the densification begins to decrease in the initial stage (up to 1%) then densification starts to increase after 1% of reinforcement. Maximum density was made for the 2% of Y_2O_3 reinforced composite. The densification behavior again starts to decrease after the reinforcement of 2% and this trend continued up to 3% of reinforcement. This behaviour was observed for all the sintering temperatures such as 1200, 1300 and 1400°C. Sintering temperatures made a remarkable influence on the densification behavior on both alloy and composites. The highest densification was achieved in the samples of sintered at 1400°C. This trend was observed in all the samples. During the

sintering process, the density increases due to the mechanism of movement of particles with high speed and grain boundary development along the pores. The experiment of making Ti6Al4V/ Y_2O_3 composite through conventional methods of powder metallurgy was done and concluded that the higher densification (3.9 g/cm^3 and 87.69% of relative density) was achieved in Ti6Al4V/ 2% wtY_2O_3.

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